PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT	То:
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(PCT Rule 61.2)	Office (Box PCT)
	Crystal Plaza 2 Washington, DC 20231
Date of mailing (day/month/year)	ÉTATS-UNIS D'AMÉRIQUE
21 July 1999 (21.07.99)	in its capacity as elected Office
International application No. PCT/US98/21287	Applicant's or agent's file reference 6010-6522
International filing date (day/month/year)	Priority date (day/month/year)
08 October 1998 (08.10.98)	09 October 1997 (09.10.97)
Applicant JONES, Gregory, M. et al	
oonzo, orogory, writer	
The designated Office is hereby notified of its election made	de:
X in the demand filed with the International Prelimina	ry Examining Authority on:
23 April 1999	(23.04.99)
in a notice effecting later election filed with the Inter	national Bureau on:
2. The election X was	
was not	
made before the expiration of 19 months from the priority Rule 32.2(b).	gate or, where Rule 32 applies, within the time limit under
Nule 32.2(b).	
The International Bureau of WIPO	Authorized officer
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(74) Agents: DUNLAP, Charles, E. et al.; Howell & Haferkamp, L.C., Suite 1400, 7733 Forsyth Boulevard, St. Louis, MO 63105 (US).

(63) Related by Continuation (CON) or Continuation-in-Part (CIP) to Earlier Application

US

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9 October 1997 (09.10.97)

(71) Applicant (for all designated States except US): BAKER HUGHES INCORPORATED [US/US]; 3900 Essex Lane, Houston, TX 77027 (US).

(72) Inventors; and

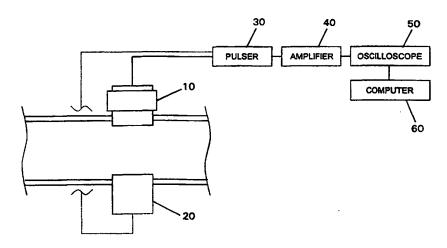
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Published

With international search report.

(54) Title: MEASUREMENT AND CONTROL OF ASPHALTENE AGGLOMERATION IN HYDROCARBON LIQUIDS



(57) Abstract

A method is provided for measuring the agglomerative state of asphaltenes in oil by applying an acoustic signal to the oil, detecting the scattered acoustic energy and using this detected signal to determine the relative particle size distribution of the asphaltene particles in the oil and/or their state of agglomeration. A method for controlling the agglomerative state of the asphaltenes which is based on the acoustic measurement technique is also provided.

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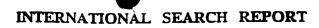
. and well BY ART 34 AMDT

- 25
- 13. A method as set forth in claim 12, wherein the signal input probe and the sensor are located so that the signal direction of the probe intersects the signal direction of the sensor at an angle of less than about 60°.
- 14. A method as set forth in claim 13, wherein the signal input probe and the sensor are located so that the signal direction of the probe intersects the signal direction of the sensor at an angle of less than about 45°.
- 15. A method as set forth in claim 1, wherein the signal of acoustic energy is applied as a pulse and the step of resolving the magnitude of the detected scattered acoustic energy at selected frequencies within the selected frequency range comprises gating the detected scattered acoustic energy to that part of the detected energy emanating from a focal region and Fourier transforming the detected scattered energy into a magnitude vs. frequency format.
- 16. A method as set forth in claim 1, wherein the signal of acoustic energy is applied as a tone-burst and the step of resolving the magnitude of the detected scattered acoustic energy at selected frequencies within the selected frequency range comprises detecting the magnitude of the scattered energy at selected frequencies within the selected frequency range.
- 17. A method as set forth in claim 1, wherein determining the agglomerative state of the asphaltenes is effected by comparing the distribution of the asphaltene particles scattering acoustic energy within the selected frequency range with a standard.
- 18. A method as set forth in claim 17, wherein the standard is a sample of known particle size.
- 19. A method as set forth in claim 17, wherein the standard is a model of particle size based on scattering theory.
- 20. A method as set forth in claim 1, wherein the oil containing asphaltenes is in a process flow stream and the signal of acoustic energy is applied to the oil in the process flow stream.
- 21. A method for measuring the agglomerative state of asphaltenes in an oil containing asphaltenes comprising:
 - a. removing a sample of the oil and without diluting the oil;

INTERNATIONAL SEARCH REPORT

ternational application No. PCT/US98/21287

	SIFICATION OF SUBJECT MATTER G01N 29/02		
	73/61.75 International Patent Classification (IPC) or to both n	ational classification and IPC	
	DS SEARCHED		
	ecumentation searched (classification system followed	by classification symbols)	
U.S. : 7	3/61.75, 64.41, 64.42, 61.71, 64.53, 53.05, 602, 599,	610, 611, 629, 865.5	
Documentati	on searched other than minimum documentation to the	extent that such documents are included	in the fields searched
Electronic da	ata base consulted during the international search (name	ne of data base and, where practicable,	search terms used)
APS search term	ns: asphaltene, agglomeration, petroleum, particle		
c. Doct	UMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where app	ropriate, of the relevant passages	Relevant to claim No.
Y	US 5,132,225 A (DICKAKIAN) 21 Ju abstract.	uly 1992 (21.07.92) see the	1-25
Y	De Boer et al, Screening of Crude Oi Theory, Practice, and the Selection of SPE Production & Facilities, pages 55-	Inhibitors, February 1995,	1-25
Y	US 4,509,360 A (ERWIN et al) 09 A lines 58+	april 1985 (09.04.85) col. 2	1-25
A	US 4,706,509 A (RIEBEL) 17 November document.	er 1997 (17.11.97) See entire	1-251
A	US 5,546,792 A (BECKER) 20 Augus document.	st 1996 (20.08.96) see entire	1-251
X Furth	er documents are listed in the continuation of Box C.	See patent family annex.	
A do	ecial categories of cited documents: cument defining the general state of the art which is not considered be of particular relevance	"T" later document published after the int date and not in conflict with the app the principle or theory underlying th	lication but cited to understand
E ear	lier document published on or after the international filing date	"X" document of particular relevance; the considered novel or cannot be considered when the document is taken alone	
cite	ed to establish the publication date of another citation or other scial reason (as specified)	"Y" document of particular relevance; the	
	cument referring to an oral disclosure, use, exhibition or other	considered to involve an inventive combined with one or more other suc being obvious to a person skilled in	ch documents, such combination
	cument published prior to the international filing date but later than priority date claimed	"&" document member of the same pater	nt family
	actual completion of the international search MBER 1998	Date of mailing of the international se	arch report
Name and a	nailing address of the ISA/US	Authorized officer	•
	ner of Patents and Trademarks	MAMICHAEL J. BROCK M. J	N. S.
1	n, D.C. 20231 Io. (703) 305-3230	Telephone No. (703) 305-4759	



emational application No. PCT/US98/21287

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	US 5,420,040 A (ANFINDSEN et al) 30 May 1995 (30.05.95) see entire document.	1-251
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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT	Article	36	and	Rule	70
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REC'D 0 2 FEB 2000

Applicant's or agent's file reference 194-10244-PCT	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No.	International filing date (day/m	
PCT/US98/21287	08 OCTOBER 1998	09 OCTOBER 1997
International Patent Classification (IPC) (IPC) and US Cl.: G01N 29/02 and	d 073/061.750	
Applicant BAKER HUGHES INCORPORATED		
Examining Authority and is	transmitted to the applicant a	been prepared by this International Preliminary according to Article 36.
2. This REPORT consists of a	total of sheets.	
been amended and are th		ets of the description, claims and/or drawings which have leets containing rectifications made before this Authority. Instructions under the PCT).
These annexes consist of a to	tal of sheets.	
3. This report contains indication	ns relating to the following ite	ems:
I X Basis of the repor	rt	
II Priority		
III Non-establishmer	nt of report with regard to no	velty, inventive step or industrial applicability
IV Lack of unity of	invention	
V X Reasoned statemer citations and expla	nt under Article 35(2) with regunations supporting such statem	ard to novelty, inventive step or industrial applicability; nent
VI Certain documents	cited	
VII Certain defects in t	he international application	
VIII Certain observation	ns on the international applicati	ion
		'
		•
Date of submission of the demand	Date	e of completion of this report
21 JULY 1999	(03 JANUARY 2000 -
Name and mailing address of the IPEA/		norized officer
Commissioner of Patents and Trader Box PCT		HEZRON E. WILLS
Washington, D.C. 20231 Facsimile No. (703) 305-3230		phone No. (703) 305-4705

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(
,	International application No.
ĺ	PCT/US98/21287

I. Basis of	the report		
			ich have been furnished to the receiving Office in response to an invitation and are not annexed to the report since they do not contain amendments):
		d application as originall	·
X	the description,	pages (See Attached)	, as originally filed.
		pages	, filed with the demand.
		pages	, filed with the letter of
			, filed with the letter of
X	the claims,	Nos. (See Attached)	, as originally filed.
		Nos	, as amended under Article 19.
			, filed with the demand.
		Nos	, filed with the letter of
		Nos	, filed with the letter of
X	the drawings,	sheets/fig (See Attached)	, as originally filed.
		sheets /fig	, filed with the demand.
		sheets /fig	, filed with the letter of
		sheets /fig	, filed with the letter of
x x x	the description, the claims, the drawings,	Nos. NONE sheets/fig NONE	· ·
	-		the amendments had not been made, since they have been considered in the Supplemental Box Additional observations below (Rule 70.2(c)).
4. Addition NONE	al observations, i	f necessary:	



International application No.

· · · · · · · · · · · · · · · · · · ·			- industrial analisability
Reasoned statement under Article 3: citations and explanations supporting			r industrial applicability;
STATEMENT			
Novelty (N)	Claims	1 - 26	YES
• • •	Claims	NONE	NO
Inventive Step (IS)	Claims	1 - 26	YE
	Claims	NONE	NO NO
Industrial Applicability (IA)	Claims	1 - 26	YE
	Claims	NONE	NO
Crude Oils for Asphalt Precipitation" [article Opinion of October 27, 1999 these reference scattered acoustic energy at selected frequent back-scattered acoustic signal from an amount same references contain therein a valid motion.	ces do not contai ncies within a se plitude versus ti tivational premis	the application's instant step of rected frequency range {i.e there e signal into a magnitude versus for combining the chromatography	esolving the magnitude of is no transformation of the sfrequency signal; nor do the only method of Dickakian with
Opinion of October 27, 1999 these reference scattered acoustic energy at selected frequent back-scattered acoustic signal from an am,	ces do not containates within a semplitude versus this tivational premiser de Boer et al. a surement and contained prior art or recordinary skill in the of all known and	the application's instant step of rected frequency range {i.e there e signal into a magnitude versus for combining the chromatographical amental guide in order to arrive ol of asphaltene agglomeration is rd}, and to manifest a high degree art at the time of the invention,	esolving the magnitude of is no transformation of the frequency signal; nor do the ohy method of Dickakian with at the instant invention-deemed to contain novelty, to be of industrial applicability even for a skilled artisan or
Opinion of October 27, 1999 these reference scattered acoustic energy at selected frequence back-scattered acoustic signal from an amy same references contain therein a valid most that acoustic method of Erwin et al. and/or hence, the claimed subject matter per meas constitute an inventive step {over the cited from the vantage point of one possessing of theoretician who was privy to wide ranges	ces do not containates within a semplitude versus this tivational premiser de Boer et al. a surement and contained prior art or recordinary skill in the of all known and	the application's instant step of rected frequency range {i.e there e signal into a magnitude versus for combining the chromatographical amental guide in order to arrive ol of asphaltene agglomeration is rd}, and to manifest a high degree art at the time of the invention,	esolving the magnitude of is no transformation of the is frequency signal; nor do the only method of Dickakian with e at the instant invention- deemed to contain novelty, to be of industrial applicability even for a skilled artisan or
Opinion of October 27, 1999 these reference scattered acoustic energy at selected frequence back-scattered acoustic signal from an amy same references contain therein a valid most that acoustic method of Erwin et al. and/or hence, the claimed subject matter per meas constitute an inventive step {over the cited from the vantage point of one possessing of theoretician who was privy to wide ranges	ces do not containates within a semplitude versus titutivational premisure de Boer et al. a surement and contained prior art or recordinary skill in the of all known and	the application's instant step of rected frequency range {i.e there e signal into a magnitude versus for combining the chromatographical amental guide in order to arrive ol of asphaltene agglomeration is rd}, and to manifest a high degree art at the time of the invention,	esolving the magnitude of is no transformation of the is frequency signal; nor do the only method of Dickakian with e at the instant invention- deemed to contain novelty, to be of industrial applicability even for a skilled artisan or
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Opinion of October 27, 1999 these reference scattered acoustic energy at selected frequence back-scattered acoustic signal from an amy same references contain therein a valid most that acoustic method of Erwin et al. and/or hence, the claimed subject matter per meas constitute an inventive step {over the cited from the vantage point of one possessing of theoretician who was privy to wide ranges	ces do not containates within a semplitude versus titutivational premisure de Boer et al. a surement and contained prior art or recordinary skill in the of all known and	the application's instant step of rected frequency range {i.e there e signal into a magnitude versus for combining the chromatograps a mental guide in order to arrive to old of asphaltene agglomeration is rd}, and to manifest a high degree art at the time of the invention, orgous prior art during that time p	esolving the magnitude of is no transformation of the frequency signal; nor do the ohy method of Dickakian with e at the instant invention-deemed to contain novelty, to be of industrial applicability even for a skilled artisan or
Opinion of October 27, 1999 these reference scattered acoustic energy at selected frequence back-scattered acoustic signal from an amy same references contain therein a valid most that acoustic method of Erwin et al. and/or hence, the claimed subject matter per meas constitute an inventive step {over the cited from the vantage point of one possessing of theoretician who was privy to wide ranges	ces do not containates within a semplitude versus titutivational premisure de Boer et al. a surement and contained prior art or recordinary skill in the of all known and	the application's instant step of rected frequency range {i.e there e signal into a magnitude versus for combining the chromatographical amental guide in order to arrive ol of asphaltene agglomeration is rd}, and to manifest a high degree art at the time of the invention,	esolving the magnitude of is no transformation of the frequency signal; nor do the ohy method of Dickakian with e at the instant invention-deemed to contain novelty, to be of industrial applicability even for a skilled artisan or

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/US98/21287

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

Sheet 10

I. BASIS OF REPORT:

This report has been drawn on the basis of the description, pages. 1-23, as originally filed. pages, NONE, filed with the demand. and additional amendments:

not applicable

This report has been drawn on the basis of the claims, numbers, 1-12 & 22-26, as originally filed. numbers. NONE, as amended under Article 19. numbers. NONE, filed with the demand. and additional amendments:

Claims 13-21 filed with the letter of 01 November 1999

This report has been drawn on the basis of the drawings, sheets, 1 - 10, as originally filed. sheets. NONE, filed with the demand. and additional amendments: NONE